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(NASA-Case-NPO-15811-1) METHOD AND  
APPARATUS FOR MINIMIZING CONVECTION DURING  
CRYSTAL GROWTH FROM SOLUTION Patent  
Application (NASA) 23 p HC A02/MF A01

N84-12968

Unclas  
44423

CSCL 20B G3/76



NASA CASE NO. NPO-15,811-1

PRINT FIG. 3

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S N. 547,175

Filed: 10-31-83

NRO-JPL

THIS NASA INVENTION APPEARS TO HAVE  
EXCELLENT COMMERCIAL POTENTIAL

AWARDS ABSTRACT

Inventor: Paul J. Shlichta      JPL Case NO. 15811  
NASA Case No. NPO-15811  
JJ&P Docket No. JET1-A65

Date: August 15, 1983  
Contractor: Jet Propulsion Laboratory

METHOD AND APPARATUS FOR MINIMIZING  
CONVECTION DURING CRYSTAL GROWTH FROM SOLUTION

The purpose of this invention is to minimize or avoid buoyant convection during solution crystallization in a gravitational field. A method and apparatus is disclosed where the crystals are grown from either the upper or lower end of a generally vertical, shallow chamber under temperature conditions that minimize the effects of buoyant convection. Growth from the upper end of the chamber is conducted under isothermal conditions. Growth from the lower end of the chamber is conducted in a positive thermal gradient.

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Contractor	Caltech/JPL
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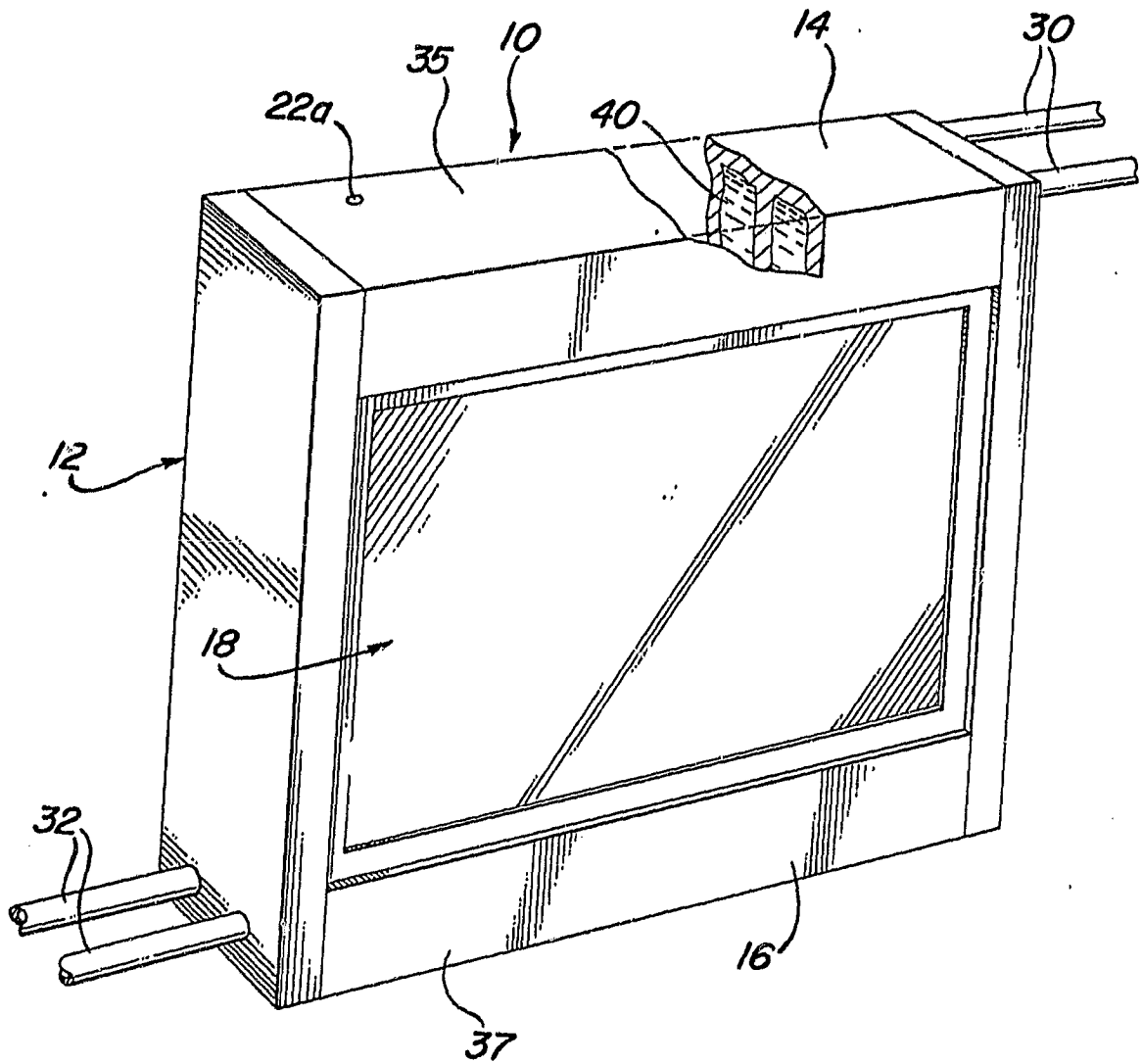


FIG. 1

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FIG. 2

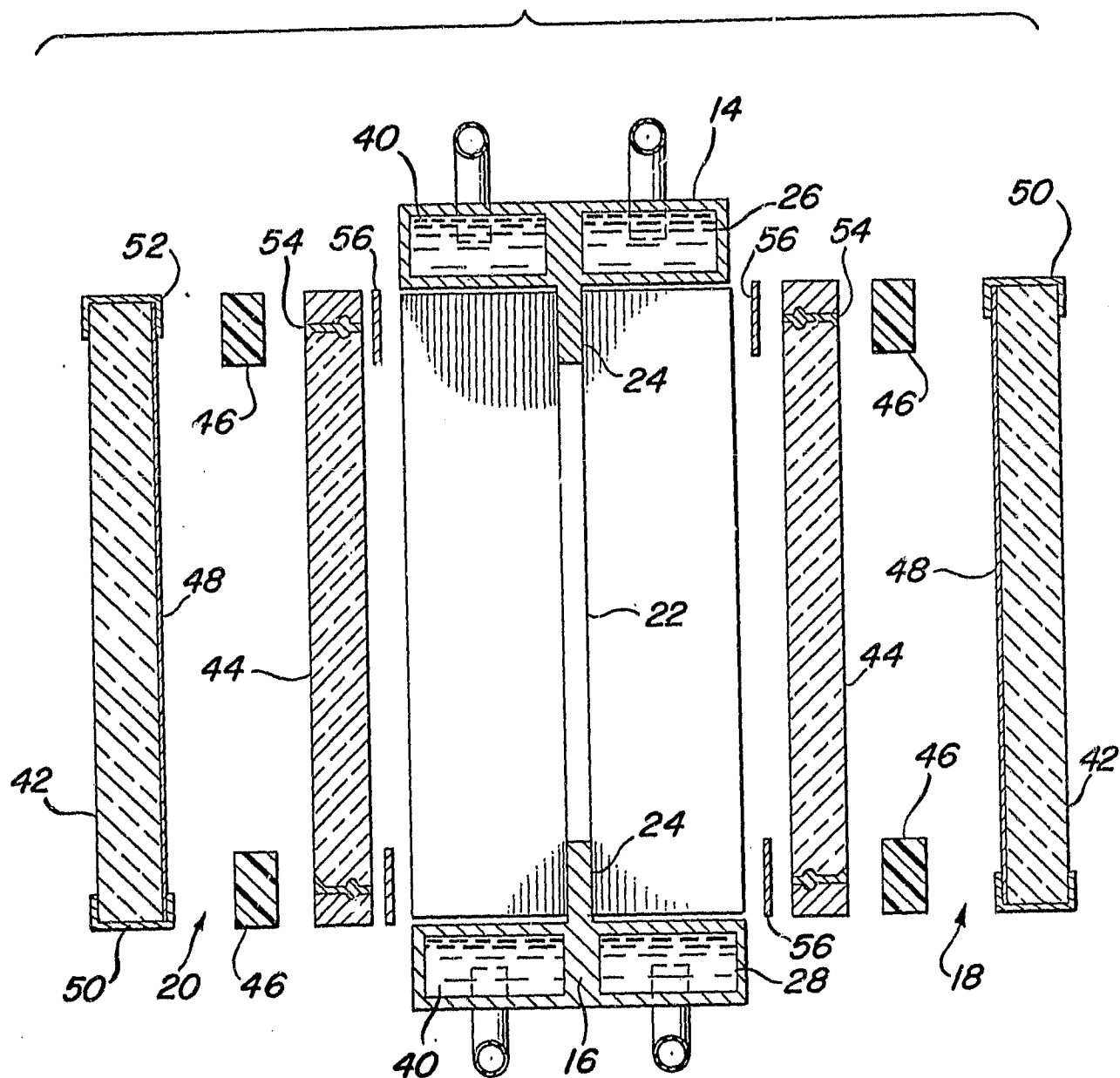


FIG. 3

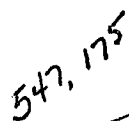
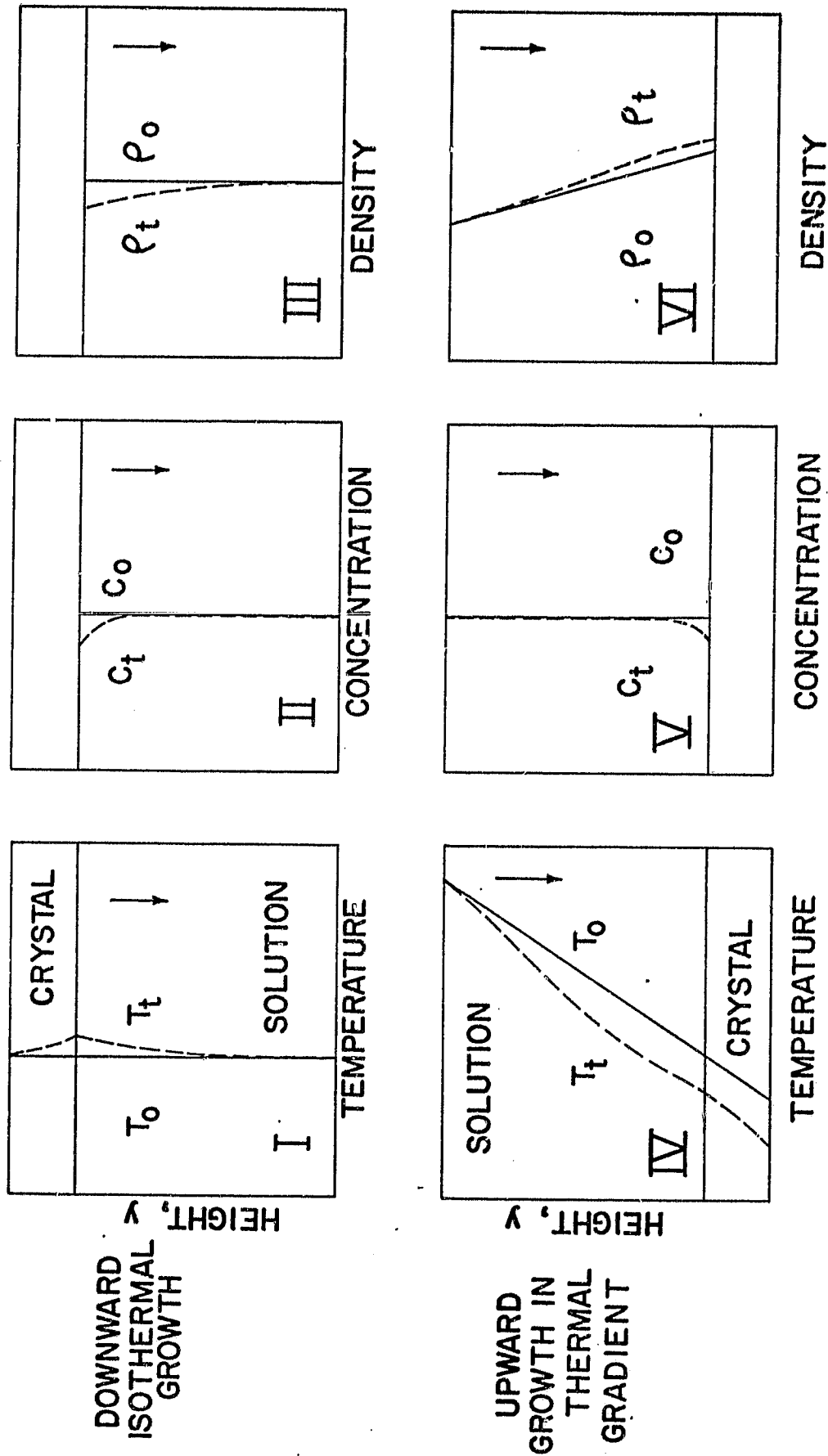


FIG. 4



$T_0, C_0, \rho_0$  - INITIAL CONDITION OF SOLUTION

$T_t, C_t, \rho_t$  - CONDITION AFTER SUBSTANTIAL GROWTH

1 JJ&P Case No.: JET1-A65  
2 JPL Case No.: 15811  
3 NASA Case No.: NPO-15811  
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8 METHOD AND APPARATUS FOR MINIMIZING  
9 CONVECTION DURING CRYSTAL GROWTH FROM SOLUTION  
10

11 BACKGROUND OF THE INVENTION

12 1. Origin of the Invention

13 The invention described herein was made in the  
14 performance of work under a NASA Contract and is subject  
15 to the provisions of Section 305 of the National Aeronau-  
16 tics and Space Act of 1958, Public Law 85-568 (72 STAT  
17 435; 43 USC 2457).  
18

19 2. Field of the Invention

20 This invention relates to a method and appa-  
21 rus for growing in a gravitational field a crystal from  
22 solution where buoyant convection is minimized. In  
23 particular it relates to a method and apparatus for  
24 growing crystals wherein the crystals are grown from  
25 either the upper or lower end of a generally vertical,  
26 shallow, chamber under temperature conditions that  
27 minimize the effects of buoyant convection.  
28

29 3. Background Discussion

30 Solid materials may be dissolved in a liquid  
31 and, as the temperature of the solution is reduced under  
32 controlled conditions, the solid materials precipitate  
33 from the solution as a single crystal. In a gravita-  
34 tional field such crystallization is normally accom-  
35 panied by buoyant convection caused by density gradients



1 in the solution near the crystal being formed. Thus the  
2 less dense or lighter portions of the solution float  
3 upwardly and the denser or heavier portions of the  
4 solution move downwardly. These density gradients are  
5 the results of differences in solute concentration  
6 within the solution surrounding the crystal. As solute  
7 is depleted with crystal growth, these concentration  
8 differences cannot be avoided.

9         Such crystallization is normally accompanied  
10 by buoyant convection caused by density gradients in the  
11 solution near the crystal being formed. Thus, the less  
12 dense or lighter portions of the solution float upwardly  
13 and the denser or heavier portions of the solution move  
14 downwardly. These density gradients are the result of  
15 differences in solute concentration within the solution  
16 surrounding the crystal. As solute is depleted with  
17 crystal growth, these concentration differences cannot  
18 be avoided.

19         There also exist temperature differences  
20 within the solution surrounding the crystal which may  
21 also produce buoyant convection. These are of secondary  
22 importance, but nevertheless must be taken into account.  
23 Since density is a function of both concentration and  
24 temperature, such temperature gradients may thus produce  
25 buoyant convection, but to a far less degree than concen-  
26 tration gradients.

27         Buoyant convection interferes with researchers'  
28 study of the mechanism of the crystal growth process.  
29 Specifically, it interferes with the measurement of  
30 temperature in concentration gradients as a function of  
31 the rate of growth. Many researchers believe that  
32 convection is also linked to or causes the formation of  
33 liquid inclusions in the crystal being formed, which is  
34 a highly undesirable result. Superior crystals which  
35 avoid such liquid inclusions may be made under zero

1 gravity conditions provided by spaceflight. Other crystal  
2 tal properties, such as purity, may also be substantially  
3 tially improved when buoyant convection is eliminated by  
4 the absence of gravity.

5 In a gravitational field, buoyant convection  
6 has only been eliminated in growing microscopic crystals,  
7 that is, crystals that can only be seen under a  
8 microscope. These microscopic crystals are of no  
9 practical value. Macroscopic crystals, those observable  
10 with the naked eye, have been grown without subjecting  
11 the crystals to buoyant convection by employing forced  
12 convection. This is accomplished by creating, under  
13 controlled conditions, currents within the solution that  
14 have a known flow pattern. For example, by stirring the  
15 solution in a prescribed way using a mounting holding  
16 the crystal. This forced convection overwhelms and  
17 mitigates the effects of buoyant convection. Creating  
18 forced convection is, however, a complex procedure, and  
19 the desired results are not always obtained.

20 Thus, a simple method and apparatus has long  
21 been desired which provides minimum buoyant convection  
22 in a gravitational field for growing a macroscopic  
23 crystal, thereby avoiding the need to grow crystals in a  
24 zero gravity environment. This invention also finds  
25 utility in a spaceflight environment which under current  
26 conditions still has a residual gravity field and  
27 transient acceleration that produces buoyant convection.

28

29

### 30 SUMMARY OF THE INVENTION

31 This invention provides a simplified method  
32 and apparatus for minimizing buoyant convection during  
33 crystal growth from solution in the presence of a gravitational  
34 field. The central feature of this invention  
35 is to establish conditions within a chamber where a

1 macroscopic crystal is grown so that the density  
2 gradients caused by concentration changes within the  
3 solution due to crystal growth are offset to avoid or  
4 minimize buoyant convection. Specific conditions are:  
5 (a) the chamber in which the macroscopic crystal is  
6 grown is generally boxlike, but very shallow in the  
7 depth dimension, so that crystal growth is essentially  
8 unidirectional along the vertical, (b) the temperature  
9 of the solution is controlled to either alter the  
10 density gradient within the solution or offset the  
11 secondary effects of density gradients due to tempera-  
12 ture changes produced by crystallization, and (c) the  
13 cooling rate of the solution is slow, typically less  
14 than 1°C per hour.

15 In broad terms, the method comprises the steps  
16 of:

17 (a) holding the solution in a vertical  
18 chamber which is relatively thin, this thin dimension  
19 being generally perpendicular to the vertical,

20 (b) disposing in the chamber at one end a  
21 substrate crystal from which the crystal grows, and

22 (c) controlling the temperature conditions in  
23 the solution so that the crystal forms, and as the  
24 crystal forms, the effects of buoyant convection within  
25 the solution are minimized.

26 The design of the chamber is critical for the  
27 practice of the method of this invention. The chamber  
28 has a generally boxlike internal configuration with the  
29 depth of the chamber being relatively shallow. In  
30 general, this depth should be no greater than about  
31 8 millimeters. This type of chamber will produce a  
32 crystal which is relatively thin but of sufficient  
33 height and width so that it will constitute a macro-  
34 scopic crystal.

1           In accordance with an important feature of  
2 this invention, the substrate crystal is disposed along  
3 essentially the entire upper or entire lower end of the  
4 chamber. The macroscopic crystal grows outwardly from  
5 the substrate crystal, generally in a single plane in  
6 one direction along the vertical axis of the chamber.  
7 The use of such a thin chamber restricts the direction  
8 of buoyant convection to a plane perpendicular to the  
9 thin dimension. This greatly facilitates the suppression  
10 of secondary convection due to thermal gradients as  
11 described below.

12           When the crystal is disposed at the upper end  
13 of the chamber, the temperature of the solution is maintained  
14 essentially isothermal. This will offset any of  
15 the secondary effects of density changes due to fluctuations  
16 in temperature. As the crystal grows downwardly  
17 from the upper end of the chamber, the natural conditions  
18 occurring within the solution in the chamber  
19 provide a less dense or lighter solution overlying a  
20 denser or heavier solution. Under these conditions  
21 there is no tendency for buoyant convection to occur,  
22 since the lighter solution is above the heavier solution.  
23 It is when this condition is reversed, that is,  
24 heavier solution over lighter solution that buoyant  
25 convection occurs.

26           When the crystal is grown from the lower end  
27 upwardly, the temperature conditions are adjusted so  
28 that the temperature of the solution in the upper  
29 portions of the chamber is substantially higher than the  
30 temperature of the solution at the interface where  
31 crystal growth occurs. Because of this temperature  
32 differential, even though the solution in the upper  
33 portions of the chamber would under isothermal conditions  
34 be heavier than solution at the interface, the  
35 decrease of solution temperature with increasing

1 temperature offsets this and produces a less dense  
2 solution in the upper portions of the chamber even  
3 though the concentration of solute in the upper portions  
4 is substantially higher than at the interface. This  
5 condition is referred to herein as a "positive thermal  
6 gradient." That is, the temperature of the solution  
7 increases with distance from the interface where crystal  
8 growth occurs so that the density of the solution in the  
9 upper regions of the chamber is less than in the lower  
10 regions. Typically, this temperature difference may be  
11 as high as 30°C or higher.

12 In accordance with another aspect of this  
13 invention, an apparatus is provided for carrying out the  
14 method of this invention. This apparatus includes a  
15 pair of conductive blocks holding space insulating  
16 plates, for example, glass window panes. The blocks and  
17 plates are arranged to provide the chamber, which has a  
18 generally boxlike interior. The depth dimension of the  
19 chamber is generally perpendicular to the vertical and  
20 is substantially less than the other dimensions of the  
21 chamber. The blocks have within them channels through  
22 which a thermal fluid is circulated. Temperature  
23 sensors are employed, one to measure the temperature of  
24 the thermal fluid, the other to measure the temperature  
25 of the solution within the chamber. Means connected to  
26 the temperature sensors provide control of the tempera-  
27 ture of the solution. This apparatus may be used to  
28 grow crystals from either the upper or lower end of the  
29 chamber.

#### 30 31 BRIEF DESCRIPTION OF THE DRAWINGS

32 The features of the present invention can best  
33 be understood by reference to the following description  
34 taken in connection with the drawing where like numerals  
35 indicate like parts.

1           Figure 1 is a perspective view of the crystal-  
2 lization cell of the apparatus of this invention.

3           Figure 2 is an exploded view, in cross-  
4 section, of the crystallization cell employed in the  
5 apparatus of this invention.

6           Figure 3 is a schematic diagram of the  
7 apparatus of this invention.

8           Figure 4 is two series of graphs, one of which  
9 depicts what occurs during downward growth of the  
10 crystal and the other of which depicts upward growth of  
11 the crystal.

#### 12 13 DETAILED DESCRIPTION OF THE DRAWINGS

##### 14 Apparatus

15           Figures 1 through 3 show the apparatus 10 of  
16 this invention for growing crystals in a normal  
17 gravitational field. As best shown in Figure 3, the  
18 apparatus 10 employs a crystal growing cell 12 which  
19 includes a pair of spaced-apart copper or aluminum  
20 blocks 14 and 16 which retain "thermal pane" type  
21 windows 18 and 20. The space between the blocks 14 and  
22 16 and windows 18 and 20 form the crystal growing  
23 chamber 22. The chamber 22 is so designed that it is a  
24 relatively thin chamber so that unidirectional planar  
25 crystal growth occurs within it. Each block 14 and 16  
26 includes an inwardly pointing fin 24 which defines the  
27 depth of the chamber, with these fins separating the  
28 windows. Each block 14 and 16 also includes U-shaped  
29 channels 26 and 28, respectively, with a pair of  
30 conduits 30 and 32 extending from opposed ends of the  
31 channels. Conduits 30 and 32 extend outwardly from,  
32 respectively, blocks 14 and 16, and place the channels  
33 in communication with thermal fluid circulators 34 and  
34 36, respectively. These circulators include heaters  
35 (not shown) for adjusting the temperature of the thermal

1 fluid. A pump (not shown) in the circulators 34 and 36  
2 pumps thermal fluid 29 through the conduits 30 and 32  
3 into and out of the channels 26 and 28 in each block.  
4 The sides 35 and 37 (Figure 1) of the cell 12 may be  
5 insulated and equipped with auxiliary heaters.

6 As best shown in Figure 2, each window 18 and  
7 20 comprises two plates 42 and 44 of glass which are  
8 spaced apart by means of a pair of Lucite spacers 46.  
9 The outer plates 42 are held in nickel-plated steel  
10 frames 52. At the edges of the inner plates 42 is an  
11 epoxy seal 54. A paraffin gasket 56 is provided between  
12 the inner window and the edges of the blocks 14 and 16  
13 and serves as a seal. The outer plate 42 of glass has  
14 its inside surface coated with a transparent conductive  
15 film 48. This film may include several separate seg-  
16 ments each maintained at a different temperature than  
17 the others. This would be the case when the macroscopic  
18 crystal is grown from the lower end of the chamber 22.  
19 The outer plates are held in electrically conductive  
20 frames 50.

21 As shown in Figure 1, when assembled, the  
22 inner and outer plates 42 and 44 of each window provide  
23 a thermal pane type of structure. The inner plates abut  
24 the fins 24, and the fins 24 serve as spacers defining  
25 the smallest dimension of the chamber 22. This smallest  
26 dimension is generally perpendicular to the vertical  
27 growth direction of the crystal in the chamber 22. The  
28 assembled structure will provide a crystallization  
29 cell 12 providing a chamber 22 having a boxlike  
30 interior. The typical dimensions of the chamber 22 are  
31 75 x 75 x 4 millimeters.

32 As shown in Figure 3, two thermocouples 56 and  
33 58 are disposed in the cell 12. One is disposed in the  
34 channel 26 of the upper block 14 and the other is  
35 disposed adjacent the inside surface of the outer glass

1 plate 44. These two thermocouples 56 and 58, serving as  
2 temperature sensors, are connected to a temperature  
3 controller 60. This controller 60 regulates the current  
4 flowing into the film 48 covering the surface of one of  
5 the plates 42.

6 The cell 12 of the above design provides a  
7 "thin" chamber 22 for holding the solution from which  
8 the crystal is grown. This chamber design "two dimen-  
9 sionalizes" the convection pattern within the cell 12  
10 and the thermal pane type windows 18 and 20 minimize the  
11 heat flow from or into the chamber 22. The thermal  
12 fluid temperature is regulated to provide the desired  
13 temperature conditions within the chamber 22 for crystal  
14 growth 22.

#### 15 16 Method

17 The apparatus 10 is used to carry out the  
18 method of this invention for growing macroscopic  
19 crystals with minimum buoyant convection. In accordance  
20 with the method of this invention, the crystal may be  
21 grown from either the upper end of chamber 22 or the  
22 lower end of chamber 22. As shown in Figure 1, the  
23 solution containing the crystal material is introduced  
24 into the chamber 22 by means of a hypodermic needle (not  
25 shown) which is inserted through a small diameter  
26 hole 22(a) in the block 14.

27 When the crystal is grown from the top down, a  
28 substrate crystal is first mounted on the edge of the  
29 upper fin 24 prior to assembly of the apparatus. Next,  
30 the solution is introduced into the chamber 22 at a  
31 temperature which avoids dissolving the substrate  
32 crystal, and the circulators are programmed so that  
33 there is essentially no temperature difference between  
34 the thermal fluid flowing through the upper and lower  
35 blocks 14 and 16. Since conditions are not at a steady



1 state, this must be taken into account. Thus, the  
2 temperature control for the transparent film 48 is set  
3 so that the difference in temperature between the window  
4 and the thermal fluid flowing through the upper block  
5 will provide a higher temperature at the surface of the  
6 windows 18 and 20 than the solution of the material in  
7 the chamber. Consequently, enough heat is introduced  
8 into the chamber to compensate for heat losses. Thus,  
9 these windows will act as quasi-adiabatic windows and  
10 prevent the flow of heat either into or from the  
11 chamber 22.

12 As crystal grows downwardly in a vertical  
13 direction, solute material in the solution is removed,  
14 thus lowering the temperature at the interface between  
15 the solid crystal and the solution. Because of the  
16 essentially isothermal temperature conditions being  
17 maintained, however, this tends to suppress any convec-  
18 tion which would otherwise occur. As the crystal grows  
19 downwardly, these convection currents are continued to  
20 be suppressed or minimized, thus avoiding the adverse  
21 consequences of convection during crystal formation due  
22 to temperature differences.

23 In accordance with the alternate embodiment of  
24 this method, the crystal is grown from the bottom of the  
25 chamber upwardly in a vertical direction. In a similar  
26 manner the chamber is precharged with solution and the  
27 substrate crystal is mounted on the edge of the lower  
28 fin 24. In this instance, however, the temperature of  
29 the thermal fluid flowing through the upper block is  
30 substantially higher than the temperature of the thermal  
31 fluid flowing through the lower block. Again, the  
32 temperature of the windows is maintained under condi-  
33 tions which prevent the flow of heat from the chamber  
34 outwardly through these windows. As solute material is  
35 removed from the solution with the formation of crystal,

1 the density of the solution at the interface between the  
2 solid crystal and the solution decreases. However, since  
3 the temperature of the solution above the interface zone  
4 is higher, the density of this solution is less than or  
5 equal to the density of material at the interface.  
6 Temperature conditions are adjusted and the circulators  
7 are programmed so that such a positive thermal gradient  
8 is maintained continually throughout the course of the  
9 formation of the crystal. This temperature condition is  
10 adjusted carefully so that the density of the solution  
11 in the upper part of the chamber 22 is always slightly  
12 lower than or equal to the density of the solution at  
13 the interface.

14 Figure 4 illustrates graphically the tempera-  
15 ture, concentration, and density conditions under both  
16 downward isothermal growth and upward growth in a posi-  
17 tive thermal gradient. The symbols  $T_0$ ,  $C_0$ ,  $\rho_0$ , respec-  
18 tively, indicate the temperature, concentration, and  
19 density conditions at startup. The symbols  $T_t$ ,  $C_t$ ,  $\rho_t$ ,  
20 indicate respectively, temperature concentration and  
21 density conditions after substantial growth of the  
22 crystal.

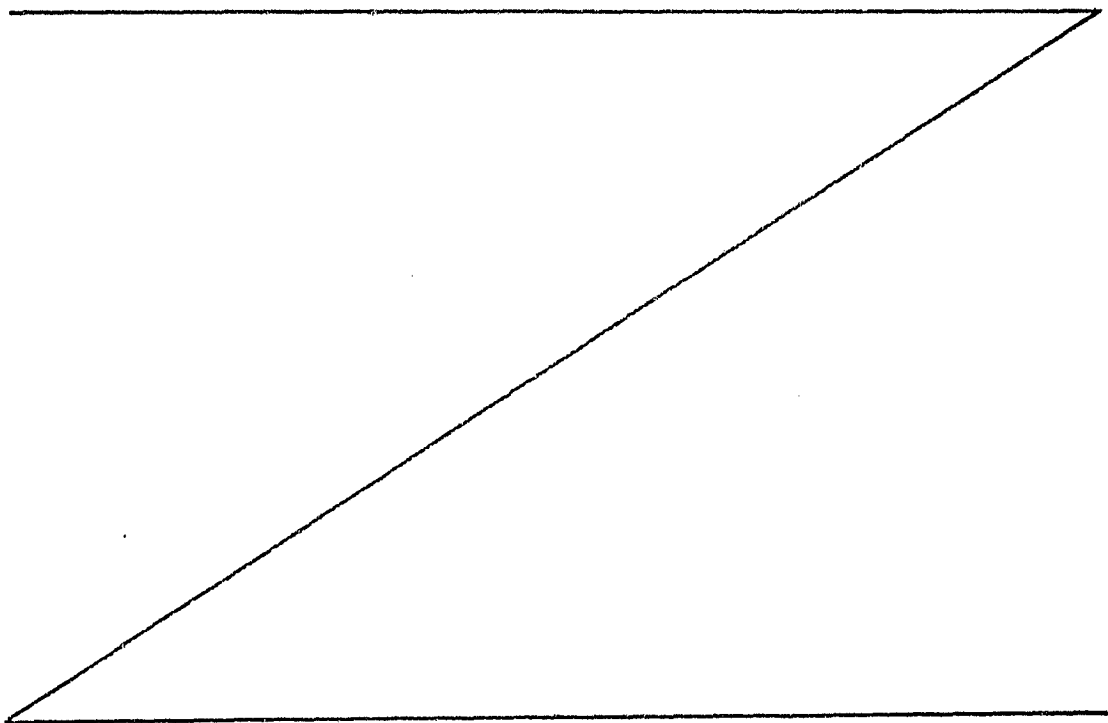
23 The three graphs marked respectively as I, II  
24 and III depict respectively the temperature, concentra-  
25 tion, and density conditions of the crystal during  
26 downward isothermal growth. As illustrated in Graph I,  
27 the initial temperature condition  $T_0$  is measured along  
28 the vertical axis of the chamber designated as y. Thus,  
29 there is initially an essentially uniform temperature  
30 throughout the entire height of the solution in the  
31 chamber 22. As the crystal begins to grow, there is in  
32 this case a slight change in temperature as the crystal  
33 forms in the upper part of the chamber. Referring to  
34 Graph II, there is initially a uniform concentration of  
35 solute in the solution which, as the crystal grows,

1 decreases at the interface between the solution and the  
2 crystal. This decrease in solute concentration would  
3 produce a condition where a lighter solution would  
4 overlay a heavier solution. This condition is depicted  
5 in Graph III. Under these conditions buoyant convection  
6 will be eliminated or minimized. Thus, growth of the  
7 crystal in the downward vertical direction inherently  
8 produces conditions which minimize buoyant convection.  
9 Again, because the chamber is relatively thin, the  
10 growth is unidirectional downwardly in a vertical  
11 direction along the entire width of the substrate  
12 crystal disposed at the upper end of the chamber 22.

13         The conditions prevalent when the macroscopic  
14 crystal is grown from the bottom end of the chamber are  
15 depicted by the Graphs IV, V and VI. As the crystal is  
16 grown from the bottom up, the thermal gradient along the  
17 vertical axis of the chamber 22 is adjusted so that the  
18 temperature of the solution will gradually increase.  
19 Accordingly, the uppermost portion of solution is at the  
20 highest temperature and the lowermost portion of the  
21 solution interfacing the crystal is at the lowest  
22 temperature. As the interface is cooled to promote  
23 crystal growth, the temperature differential will  
24 increase as indicated by the dashed line. As depicted  
25 in Graph V, the initial concentration of the solute in  
26 the solution is uniform throughout the entire solution.  
27 When crystal begins to form, this concentration decreases  
28 as indicated by the dashed line. A decrease in concen-  
29 tration would mean that a lighter or less dense solution  
30 is underlying a more dense or heavier solution. This  
31 would normally create buoyant convection. However,  
32 because of the thermal gradient applied along the  
33 vertical axis of the chamber, the temperature of the  
34 solution in the upper portions of the chamber is sub-  
35 stantially higher than those in the lower portions of

1 the chamber, producing in effect a lowered density  
2 solution overlying a less dense solution. This is  
3 illustrated in Graph VI, which shows the initial density  
4 in solid lines and the density after substantial growth  
5 of the crystal in dashed lines. Note that the least  
6 dense solution overlies the more dense solution. This  
7 density gradient suppresses buoyant convection. More-  
8 over, it is believed that growing a crystal into a  
9 solution which is at a higher temperature than at the  
10 interface provides improved crystal properties.

11 Experimentally, three different materials were  
12 used to form crystals in accordance with the method of  
13 this invention. The materials used were potassium  
14 bromide, aluminum potassium sulfate, and potassium  
15 dihydrogen phosphate. These materials were dissolved in  
16 water to form the solution and crystals were then grown  
17 from this solution in accordance with the methods  
18 discussed above. Tables 1 and 2 set forth the data  
19 collected in carrying out the experiments.



-14-

TABLE I: Isothermal mode experiments<sup>a</sup>

EXPERIMENT #	CRYSTAL	T <sub>sat</sub>	T <sub>cell</sub>	COOLING RATE	TIME	MODES OF OBSERVAT.	ONSET OF CONVECT.	T	TOTAL GROWTH	INTERFACE PROFILE	SURFACE TEXTURE	LAYERS	INCLUSIONS
233-38	Kbr(100)	50°C	50-40°C	0.42°C/hr.	25 hr	S	48°	48°C	CRYSTAL NOT SAVED FOR STUDY				
948-26	Alum(111)	"	50-43	"	17	SM	none	49°	"	"	"	"	"
948-43	"	"	50-41	"	21	SM	"	49°	"	"	"	"	"
948-64	"	"	50	"	0	SM	"	50 <sup>b</sup>	"	"	"	"	"
948-72	"	"	50-40	"	23	SM	"	49°	"	"	"	"	"
948-75	"	"	50-42	"	19	SM	"	49°	"	"	"	"	"
951-3	"	"	50-41	"	21	SM,CM	"	"	0.8mm	flat <sup>c</sup>	facet	clear, few	no
953-15	"	"	53-42	"	20	SM,CM	"	"	0.5	flat <sup>c</sup>	facet	clear, few	yes
948-86	KDP(001)	47°	47-27	"	47	SM	none	none	0.6	slightly convex <sup>c</sup>	caps & pits		
948-96	KDP(100)	49	49-39	"	24	SM,CM	Intermitt. at onset	45°	0.3	flat <sup>c</sup>	facet <sup>d</sup>	clear	
953-11	"	"	49-39	"	24	SM,CM	"	"	0.2	concave <sup>c</sup>	stepped	clear	no

NOTES: a. All experiments were carried out in the UGC4 cell with the top 0.5°C hotter than the body throughout.

b. Crystallite fell from seed crystal during filling with solution.

c. Facet along edge of crystal.

d. Plateau above location of spurious crystals.

TABLE II: Gradient mode experiments<sup>a</sup>

EXPERIMENT #	CRYSTAL	T <sub>sat</sub>	T <sub>bot</sub>	COOLING RATE	TIME	MODES OF OBSERVAT.	ONSET OF CONVECT.	TOTAL GROWTH	INTERFACE PROFILE	SURFACE TEXTURE	DENSITY OF INCLUSIONS	LAYERS
489-12(end)	Kbr(100)	53°	5°-40°C	0.42°C/hr	45 hr	S, I, S	none obs.	0.4mm.	convex <sup>b</sup>	fairly smooth	opaque	yes
489-55	"	"	55-25	"	80	S, I, S	"	2.9	"	rough	ops/clear/cellular	"
233-53	"	"	53-31	"	50	CM	"	1.1 <sup>c</sup>	"	stepped	opaque	"
233-63	"	"	51-30	"	50	SM	48°C	1.4	"	fairly smooth	opaque	"
233-81	"	"	51-31	"	50	SM(over-exposed)	a lot at end	1.0	slightly convex	fairly smooth	cloudy	"
233-86(end)	"	"	53-34	"	40	SM, I (burnout)	"	1.3	double bow <sup>d</sup>	smooth, pitted	opaque	no <sup>d</sup>
948-19	"	"	53-36	0.083	264	CM	"	0.8	sl. dbl. bow	smooth	opaque	"
948-50	"	"	53-39	"	172	SM	none obs.	0.9	"	smooth	opaque	"
948-92	KDP(001)	47	48-34	"	165	none	"	0.2	slightly convex	fairly smooth <sup>e</sup>	cloudy	N.A.

- NOTES: a. All experiments were carried out in the UGC3 cell with the top temperature at 80°C throughout.  
b. Convex or bowed profile suggests stable low-velocity convection cells.  
c. Total growth on top and bottom of seed crystal.  
d. Layering not observed in inclusion structure but may be revealed by irradiation coloring.  
e. Sharp (101) facet observed along edges of crystal.

1           The above description presents the best mode  
2 contemplated in carrying out the present invention.  
3 This invention is, however, susceptible to modifications  
4 and alternate constructions from the embodiments shown  
5 in the drawing and described above. Consequently, it is  
6 not the intention to limit the invention to the par-  
7 ticular embodiments disclosed. On the contrary, the  
8 invention is to cover all modifications and alternate  
9 constructions following within the spirit and scope of  
10 the invention as expressed in the appended claims.

METHOD AND APPARATUS FOR MINIMIZING  
CONVECTION DURING CRYSTAL GROWTH FROM SOLUTION

ABSTRACT OF THE DISCLOSURE

Disclosed is a method and apparatus for growing in a gravitational field a microscopic crystal from a solution. Solution is held in a vertical chamber which is relatively thin, the thin being generally perpendicular to the vertical. There is a substrate crystal disposed at either the upper or lower end of the chamber and the crystal grows from this substrate crystal in one direction. In accordance with this invention, the temperature conditions of the solution are controlled so that as the crystal forms the effects of buoyant convection within the solution are minimized. This is accomplished in two different ways depending upon whether the crystal is grown from the upper or lower end of the chamber. When grown from the upper end of the chamber, the temperature of the solution is controlled so that it remains essentially isothermal so that there is essentially no heat loss from the solution. When the crystal is grown from the lower end of the chamber, the temperature of the solution is controlled so that there is a differential in temperature throughout the solution which provides a positive thermal gradient within the chamber.